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Synthesis and properties of azo dye aligning layers for liquid crystal cells

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The synthesis and properties of azo dyes that can be used for photoaligning liquid crystals (LCs) have been investigated. The structures and the synthetic procedure for the azo dyes are presented. The photoaligning of azo dyes takes place purely due to the reorientation of the molecular absorption oscillators perpendicular to the UV light polarization. The qualitative model for the phenomenon in terms of the rotational diffusion of the azo dye molecules in the field of the polarized light is discussed. The order parameters $S = -0.4$ (80% of the maximum absolute value $S_m = -0.5$) were measured from the polarized absorption spectra at the wavelength 372 nm. A temperature stable pretilt angle of 5.3° was obtained by a two-step exposure of the azo dye film using normally incident polarized light followed by oblique non-polarized light. The azimuthal anchoring energy of the photoaligned substrate was $A_\phi \approx 10^{-4} \text{ J m}^{-2}$, which is the same as the anchoring of the rubbed polyimide (PI) layer. The voltage holding ratio value of a photoaligned LC cell was found to be even higher than for a rubbed PI layer, which enables the applications of azo dyes as aligning layers in active matrix liquid crystal displays. The thermal stability of the photoaligned azo dye layers is sufficiently high, but UV stability has to be improved, e.g. by polymerization. A new LCD aligning technology based on polymerized azo dye layers is envisaged.

1. Introduction

The phenomenon of photoalignment and new photoaligning materials (PAMs) has been a subject of extensive research since the effect was first described for the reversible *cis-trans*-isomerization of azobenzene units attached to a substrate [1] or the photochemical reaction of cross linking in polyvinyl 4-methoxycinnamate (PVMC) [2]. Photo-alignment is a non-contact method, which enables the creation of a high quality of orientation of a liquid crystal (LC) layer without mechanical damage to the substrate, electrostatic charge or dust contamination. Multi-domain configurations for fine pixel structures of a LC cell can be easily obtained resulting in wide viewing angles for the liquid crystal display (LCD) [3]. Photo-aligning is especially helpful for high resolution LCDs, e.g. with a liquid crystal on silicon (LCOS) structure, where normal rubbing techniques can hardly be applied [4]. After the appearance of the first photoaligned films,

such as dye doped polymer layers [5] or PVMC the development of new PAMs has become an important topic of research [6].

The substances developed can be classified by the physical origin of the photoaligning phenomenon. One of them is a photochemical reaction such as crosslinking in cinnamate polymer derivatives [2, 7–11] and another is photodegradation in photosensitive polyimides [12–14]. The quality of the photoaligned cells based on photochemical reactions proves to be very high [6]. However, such disadvantages as a comparatively low order parameter with a maximum value critically dependent on the illumination energy, the chemical nature of the PAM and the formation of by-products that may contaminate the LC material are still not tolerable. The other class of PAMs is based on the reversible effect of *cis-trans*-isomerization. A command surface containing azobenzene units was a first example [1], but the azimuthal degeneration of the LC alignment and an unstable *cis*-form stimulated the search for the new materials [15–21].

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Later polymers with azobenzene side groups [15–19], *cis-trans*-isomerizing azo-dyes contained in the polymer matrix [5] and pure dye layers [20, 21] were proposed.

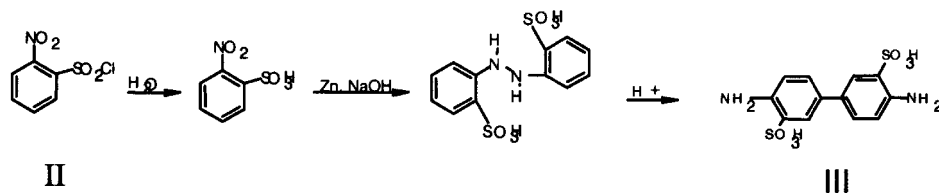
Recently we have shown the possibility of obtaining non-contact photoalignment layers using photochemically stable azo dye structures [22, 23]. Photochemically stable azo dye layers provide an example of pure reorientation of the absorption oscillator of the azo dye molecules perpendicular to the polarization of the activating UV light. As was suggested earlier by one of the authors [24], photochemical mechanisms, such as crosslinking, photodegradation and even *cis-trans*-isomerization are most probably avoided in the case of certain organic materials such as the photochemically stable azo dye layers in our case. The negligible contribution of *cis-trans*-isomerization in the photoalignment by certain amino-azobenzene molecular films has also been observed [25].

In this paper we provide the synthetic procedure for water soluble sulphonic azo dyes, together with some important parameters of the LC cells produced on the basis of the photoaligned azo dye layers, such as pretilt angle, azimuthal anchoring energy and voltage holding ratio (VHR). The model of rotational diffusion of the azo dye molecules in the field of the polarized UV light is discussed. The new photoalignment azo dyes have been shown to be temperature stable.

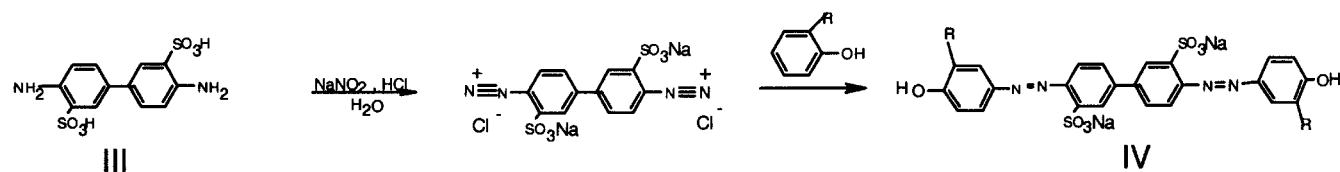
2. Experimental procedure

2.1. Synthesis of azo dyes

The sodium salt of 4,4'-bis(4-hydroxy-3-carboxy-phenylazo)benzidine-2,2'-disulphonic acid (SD1) (**I**, figure 1) was previously synthesized from 4,4'-diaminobiphenyl-2,2'-disulphonic acid prepared from 4,4'-diaminobiphenyl (benzidine) [26].



Scheme 1.



IVa: $R = -\text{COOH}$ (SD)

IVb: $R = -\text{CF}_3$ (SD2)

Scheme 2.



Figure 1. Sulphonic acid water soluble azo dye structure of SD1.

The use of benzidine as starting material for the production of water-soluble dyes has been largely discontinued in view of its toxicological properties. We have synthesized diazo dyes having a structure similar to (**I**), starting from 2-nitrobenzenesulphonyl chloride (**II**), so avoiding the use of benzidine. 4,4'-diaminobiphenyl-3,3'-disulphonic acid (**III**) was therefore prepared according to scheme 1 [27].

Based on acid (**III**) we have synthesized the diazo-dyes (**IV**) using scheme 2.

The electronic spectra of the diazo dyes **I**, **IVa** and **IVb** (see figure 2) show that the dyes containing sulphonic groups in the *o,o'*-positions with respect to the azo groups (**IV**) exhibit a distinct photochromic effect relative to the derivatives of benzidine. This is probably explained by steric twisting of the dye molecule due to the proximity of the sulphonic groups to the azo bonds. It is well known that the presence of lateral substituents in the *o*-position to an azo bond increases the photochemical stability of azo dyes [28].

2.2. Synthetic procedure

Benidine-3,3'-disulphonic acid (**III**) was diazotized by adding 0.1 ml of 30% HCl to a solution of 0.55 g (0.0022 mol) of (**III**) in aqueous NaNO_2 (0.17 g, 0.0044 mol) at room temperature. On completion of diazotisation

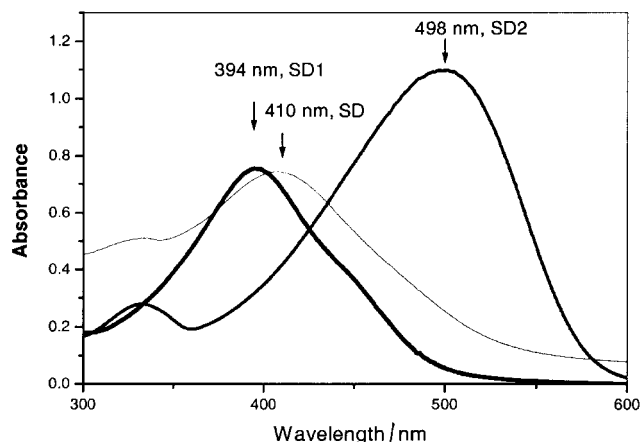


Figure 2. Electronic spectra of the azo dyes **I** (SD1), **IVa** (SD) and **IVb** (SD2) dissolved in *N,N*-dimethylformamide at a concentration of 1 wt %.

(1–2 h), the diazonium mixture was added dropwise to a solution of 0.61 g (0.0044 mol) of 2-hydroxybenzoic acid in 5% aqueous Na_2CO_3 (10 ml). On completion of coupling (7–10 h) the precipitate was filtered off and washed with a hot chloroform and acetone to give 0.85 g (60.3%) of the sodium salt of 4,4'-bis(4-hydroxy-3-carboxyphenylazo) benzidine-3,3'-disulphonic acid **IVa** (SD), $\lambda_{\text{max}} = 410$ nm, figure 2.

Diazo dye **IVb** was synthesized similarly by coupling 0.713 g (0.0044 mol) of 2-trifluoromethylphenol with the diazonium salt of benzidine-3,3'-disulphonic acid (**III**) to give 1.5 g (73.3%) of the sodium salt of 4,4'-bis(4-hydroxy-3-trifluoromethylphenylazo) benzidine-3,3'-disulphonic acid **IVb** (SD2), $\lambda_{\text{max}} = 498$ nm, figure 2. The products were purified by recrystallization and column chromatography.

2.3. Sample preparation and measurements

We checked all the azo dyes (**I**, **IVa** and **IVb**) for their usefulness in LC photoaligning and realized that the azo dye SD-1 (**I**) showed the best performance. Thus the results provided below will mostly relate to SD-1. Azo dye SD-1 was dissolved in *N,N*-dimethylformamide at a concentration of 1 wt %. The solutions were spin-coated onto glass substrates with ITO electrodes and dried at 100°C. The spin-coated film was uniform and very adhesive to the glass substrate. UV light was irradiated onto the surface of the layer using a super-high pressure Hg lamp, an interference filter at 365 nm, and a polarizing filter when required. The light intensity on the surface of the layer was 15 mW cm^{-2} for polarized light and 40 mW cm^{-2} for non-polarized light. Two glass substrates were assembled to form the liquid crystal cell with a cell gap of 10 μm . Liquid crystal mixture 11-3323 (from Dainippon Ink and Chemicals, Inc.) was injected into the cell in its isotropic phase by capillary action.

3. Experimental results

3.1. Polarized absorption spectra

In order to study the photoreorientation of the azo dye molecules (SD1) by linearly polarized UV exposure, we measured polarized absorption spectra for the layer before and after irradiation with the linearly polarized UV light. The spectra were measured using incident light with polarization directions parallel and orthogonal to the polarization direction of the activating linearly polarized UV light. Figure 3 shows the absorption spectra before (curve 1) and after (curves 2 and 3) UV irradiation.

Before irradiation, the absorption of the azo dye layer does not depend on the polarization of the light used in the measurements. After irradiation by linearly polarized UV light, the absorption of light with polarization direction parallel to the polarization direction of the activating light decreases (curve 2, figure 3), while that for an orthogonal polarization direction increases (curve 3, figure 3). The evolution of the absorption spectra after UV illumination reveals no noticeable contribution from photochemical reactions [24, 25], as the average absorption $A_{\text{ave}} = (A_{\parallel} + 2A_{\perp})/3$ remains the same for any fixed value of the exposure time, figure 3. The latter also means that we cannot detect by our spectral measurements the process of *cis-trans*-isomerization, as usually the *cis*- and *trans*-forms exist simultaneously, resulting in a corresponding variation of the average spectral absorption A_{ave} [25]. The order parameter S of the azo dye chromophores can be expressed as [29]:

$$S = (A_{\parallel} - A_{\perp}) / (A_{\parallel} + 2A_{\perp}) \quad (1)$$

where A_{\parallel} and A_{\perp} are the absorption (optical density) of the parallel and the orthogonal polarized light with respect to the polarization of the activating UV light. The order parameter S of SD1 is -0.4 at $\lambda_m = 372$ nm (absorption maximum), which is 80% of its maximum

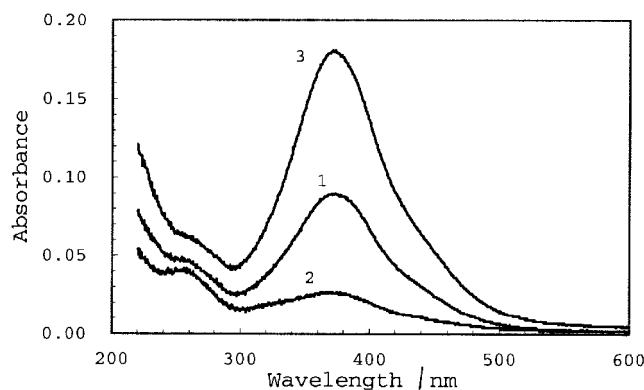


Figure 3. Absorption spectra of the SD-1 layer before exposure to polarized UV light (curve 1). Curves 2 and 3 show the polarized absorption spectra after exposure to UV light polarized in the direction parallel and perpendicular, respectively, to the polarization of the activating light.

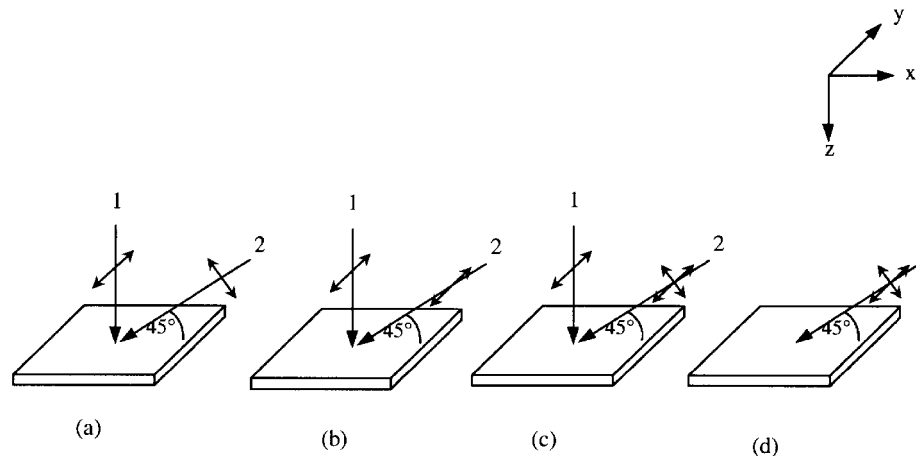


Figure 4. Various methods of UV light irradiation to attain the pretilt angle of LC layer on the photoaligned azo dye film. Case (a) two step exposure by y -polarized light vertical to the plane of the azo dye film (xy -plane) and oblique p-polarized light; (b) case (a) with s-polarized light used at the second step, (c) case (a) with non-polarized light at the second step; (d) one step exposure with oblique non-polarized light. The activating UV light propagates in the xz plane at an angle of 45° to the substrate.

absolute value $S_m = -0.5$ in our case. The dependence of the order parameter S on exposure time t_{exp} , calculated from the absorption spectra, equation (1), clearly shows that it saturates for sufficiently high values of t_{exp} .

3.2. Pretilt angle

In order to achieve pretilt of the liquid crystal molecules on the azo dye (SD1) photoaligned surface, we tried four kinds of exposure method illustrated in figure 4. To measure the pretilt angle, the liquid crystal cells were assembled with substrates antiparallel to the UV irradiation directions and they were filled with the liquid crystal mixture. The crystal rotation method was used to measure the pretilt angles [29]. Pretilt angles of 0.7° , 0.1° , 5.3° and 3.8° were obtained with exposure methods (a), (b), (c) and (d), respectively, figure 4. We believe that oblique irradiation of non-polarized light is the most efficient for obtaining large pretilt angles. The dependence of the pretilt angle on the exposure energy in method (c) is shown in figure 5. The pretilt angle was temperature stable and did not change after heating the sample to 100°C for 10 min. The SD1 photoaligned layer provides an initial LC pretilt angle value about two–three times higher than the corresponding pretilt angle for the SD2 layer.

3.3. Anchoring energy

The anchoring energy of the azo dye layer was measured according to a method already used by one of the authors for a photoaligned substrate [30]. A substrate with a rubbed polyimide alignment layer and one with a photoaligned azo dye layer were assembled with an 80° twisted nematic configuration. The resulting twist angle $\Delta\phi$ can be equal to 80° only in the case of a sufficiently high

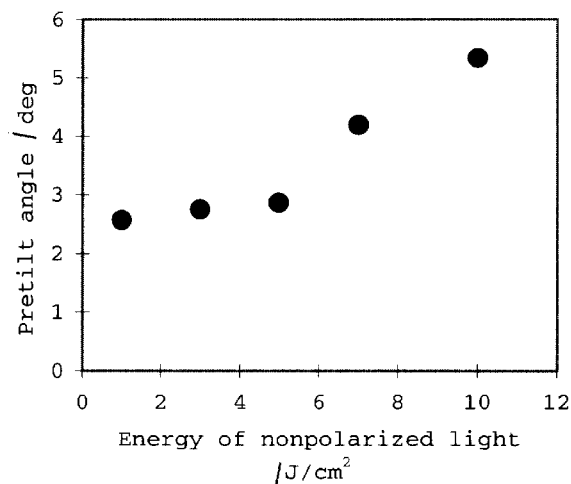


Figure 5. Dependence of the pretilt angle on the energy density of the obliquely irradiating non-polarized light after irradiation₂ by vertical polarized light of energy density 10 J cm^{-2} .

anchoring energy on the photoaligned substrate, otherwise it decreases due to the elastic torque produced by a rubbed surface where the anchoring energy is considered to be strong [29, 30]. The liquid crystal 11-3323 was injected into the cell with a gap of $10 \mu\text{m}$. The azimuthal anchoring energy of the azo dye alignment layer A_ϕ was calculated from the torque balance equation in LC cell, provided that the anchoring energy of the rubbed surface is infinite [30]:

$$A_\phi = 2K_{22}\Delta\phi/d \sin^2(\Phi - \Delta\phi) \quad (2)$$

where Φ , $\Delta\phi$, K_{22} , and d are the 80° twist angle, the twist elastic constant of the liquid crystal (K_{22} was taken

as 6.0×10^{-12} N), and the cell gap, respectively. The measured twist angle $\Delta\phi$ was about 80° , with an accuracy of 1° , for the whole range of UV light exposure energies between 1 and 10 J cm^{-2} , which says that the anchoring energy $A_\phi > 10^{-4} \text{ J m}^{-2}$, which is comparable with the anchoring energy of the rubbed polyimide layer [29].

3.4. VHR and LC electro-optical response

Figure 6 compares the transmission-voltage characteristics of a 90° twisted nematic LC cell (TN-LC) based on photoaligned azo dyes SD1, SD2 film and of a rubbed polyimide PI 2424 layer. The transmission-voltage curves (TVCs) are similar and show very good contrast, which as a result of the perfect LC alignment can be measured directly. It is greater than 70:1 at normal incidence, figure 6. The electro-optical response does not change even when the SD1 photoaligned cell is heated to 200°C during 2 h (figure 6).

The voltage holding ratio (VHR) was measured by a standard technique, when a voltage pulse $V_0 = 5 \text{ V}$ was applied during $64 \mu\text{s}$ and the voltage $V(t)$ on the LC cell dropped down during $\tau = 16 \text{ ms}$ after the pulse was switched off at $t = 0$. The VHR of the photoaligned LC cell was found to be very high (98–99%) at room temperature and $>95\%$ at $T = 80^\circ\text{C}$. The latter means, that the azo dye (SD-1) photoaligning layer cannot be a source of ions and impurities, which do not appear even at quite high temperatures ($T = 80^\circ\text{C}$) [29]. The VHR value of the photoaligned LC cell appeared to be even

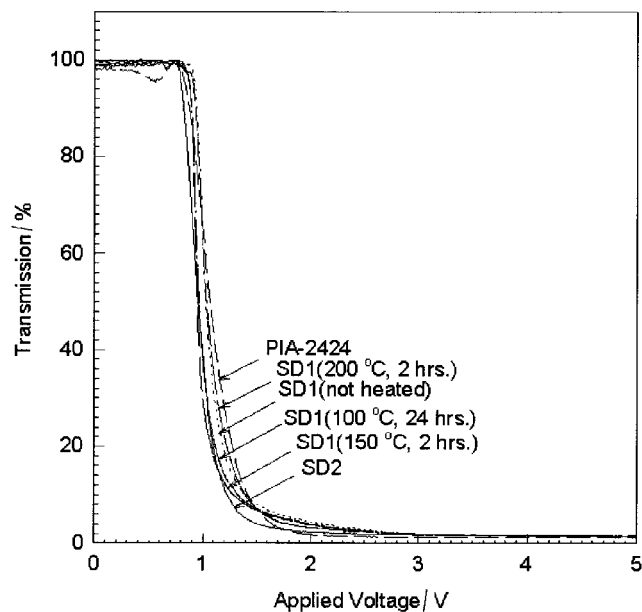


Figure 6. Comparison of transmission-voltage curves for 90° twisted nematic LC cells based on photoaligned azo dyes SD1 and SD2 film and rubbed polyimide PI 2424 layer. Heating the cell to 100, 150 and 200°C during 2 h has no effect on the curve.

larger than for the LC cell based on the rubbed polyimide layer. Thus future applications involving active matrix liquid crystal displays (AM-LCDs), where a high ionic purity of the LC cell is required, become possible.

4. Discussion

We suggest that the azo dye films are photochemically stable in our case. This was confirmed by FTIR spectra which we recorded before and after illumination by UV light of 30 J cm^{-2} , see figure 7.

The FTIR spectrum of the SD-1 layer after UV illumination does not change, testifying to the photochemical stability of our azo dye layers. However further investigations are needed to prove that the contribution of *cis-trans*-isomerization can be totally neglected in our case.

One of the possible photoaligning mechanisms in azo dye films is purely a reorientation of the azo dye molecules [23–25]. When the azo dye molecules are optically pumped by a polarized light beam, the probability of reorientation is proportional to the square of cosine θ , the angle between the absorption oscillator of the azo dye molecules and the polarization direction of the UV light, see figure 8.

Therefore, the azo dye molecules which have their absorption oscillators (chromophores) parallel to the UV light polarization will most probably acquire the increase in energy which results in their reorientation from the initial position. This results in an excess of chromophores aligned in a direction such that the absorption oscillator is perpendicular to the polarization of the UV light. In our case the chromophore is parallel to the molecular long axis of the azo dye (figure 8), i.e. the azo dye molecules tend to align with their long axes perpendicular to the UV light polarization. The function $f(\theta)$ representing the statistical distribution of the azo

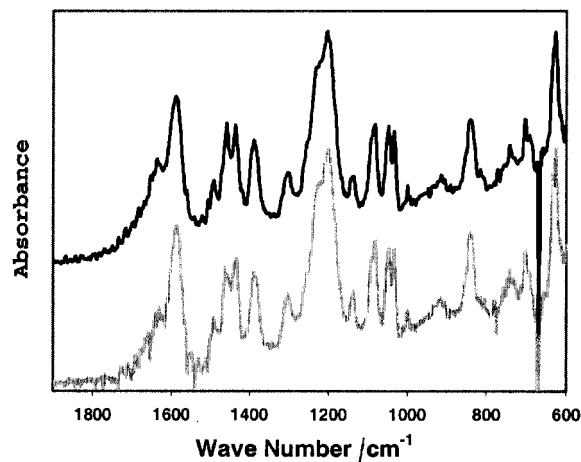


Figure 7. FTIR spectra of SD1 before (solid) and after (dotted) UV exposure with energy 30 J cm^{-2} .

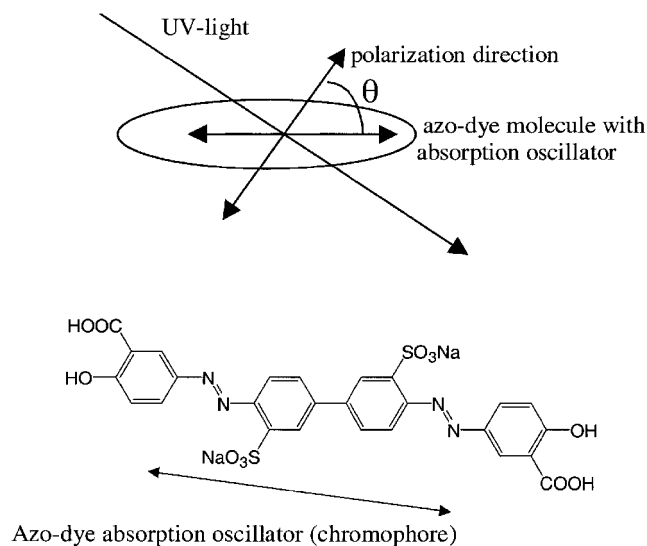


Figure 8. Qualitative interpretation of the photoinduced order in photochemically stable azo dye films. Above: the geometry of the effect; below: SD-1 molecule, having the absorption oscillator (chromophore) parallel to the molecular long axis.

dye molecules aligning along the various orientations θ , is given by $f = 1/4\pi$ in the initial state will tend to $f = \delta(\theta - \pi/2)$ for a sufficiently high exposure time. Hence, a thermodynamic equilibrium in the new oriented state will be established. Consequently, the anisotropic dichroism or birefringence is photoinduced permanently and the associated order parameter, as a measure of this effect, goes to the saturation value, which can be very large in these materials. We have shown that the order parameter S of the SD-1 layer, calculated from the polarized absorption spectra, goes to saturation for a sufficiently high exposure time [22] contrary to the case for photochemically active substances, when the order achieves its maximum value and then decreases to zero [24]. A more exact analysis of the anisotropic dichroism and photoinduced birefringence in azo dye layers based on the model of rotational diffusion will be published elsewhere.

The order parameter, induced in the UV-exposed azo dye films defines the preferred orientation of the LC molecules in the bulk, due to the dispersion interactions between the azo dye and LC molecules. Thus a new non-contact, photoalignment technique becomes possible.

5. Conclusion

We have shown that azo dye films can be used as new materials for photoalignment. We provide the synthetic procedure for the azo dye films together with their UV absorption and FTIR spectra. Based on our results, we suggest that both photochemical reactions and *cis-trans*-isomerization make no visible contribution to the photo-

induced order in azo dye films. The photoalignment phenomenon in our case can be explained by the model of rotational diffusion of the azo dye molecules in the field of the polarized UV light, whereby, in the final state, the azo dye absorption oscillators (chromophores) or molecular long axes are perpendicular to the polarization direction of the UV light.

We found, that the induced order parameter in the azo dye films is considerably high, $S = -0.4$ (80% of the maximum absolute value $S_m = -0.5$), at the wavelength of maximum absorption $\lambda = 372$ nm. A pretilt angle of 5.3° was obtained by a two-step exposure of the azo dye film using normally incident polarized light followed by oblique non-polarized light. The azimuthal anchoring energy of the photoaligned substrate was $A_\phi > 10^{-2}$ J m⁻², which is comparable with the anchoring of rubbed PI layer. The electro-optical characteristics of azo dye aligned TN LCD samples are satisfactory and the contrast ratio 70:1 can be easily obtained in photoaligned LC cells. The VHR value of the photoaligned LC cells was also found to be very high at room temperature and even at $T = 80^\circ\text{C}$.

The temperature stability of the photoaligned azo dye layers is quite high, but UV stability has to be improved. In view of this we have synthesized the SD, SD1 and SD2 molecules with polymerizable terminal groups. We have polymerized the new substances both in a solvent and as solid films, keeping the high order parameter achieved by preceding UV illumination. Our results are encouraging and will be published elsewhere.

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